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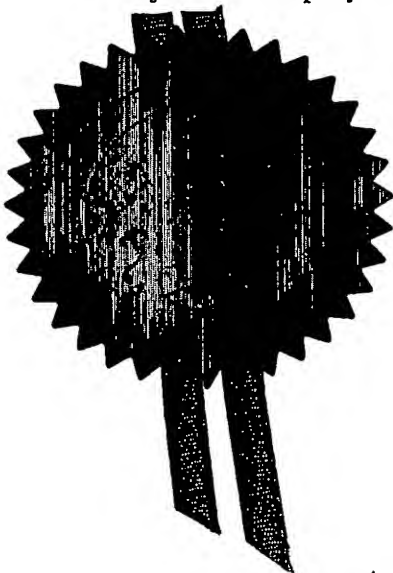
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4. Title of the invention

Composition and Method

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Patents ADP number

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7. Divisional status claimed from:

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Continuation sheets of this form -

Description

17

Claim(s)

3

Abstract

1

Drawing(s)

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Priority documents -

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Statement of inventorship and right to grant a patent (PF 7/77) -

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Request for substantive examination (PF 10/77)

1

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- 11.

We request the grant of a patent on the basis of this application.
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11 February 2004

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On behalf of the Applicant

12. Contact

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DUPLICATE

Composition and Method

This invention relates to the field of cleaning. It relates in particular to a cleaning composition, and to a
5 method of cleaning.

EP-A-330379 describes the use of cleaning compositions which contain at least one ether alcohol, water and a hydrocarbon in a single phase, respectively in the percent
10 weight ratios 90-50:2-36:25-0.9. The composition is said to be suitable for removing oil adherent from surfaces e.g. rock cuttings produced during drilling operations for oil. The compositions are made by simply mixing the
15 chemicals, hand shaking, and then allowing the mixture to separate into three phases. The middle phase was the cleaning composition, and was extracted and used for cleaning tests. This middle phase contained the ether alcohol in major proportion, and lesser amounts of water and hydrocarbon.

20

A disadvantage of this system is the large amount of alcohol ether present in the cleaning composition. The use of large amounts of many alcohol ethers is now restrained by legislation relating to VOCs (Volatile
25 Organic Components).

In accordance with a first aspect of the present invention there is provided a cleaning composition comprising at least 80% water and at least 1% of an
30 amphiphile, the composition preferably being on or adjacent to a phase boundary on the phase diagram for the composition.

In this specification when a percentage value is given for a component, it denotes the percentage by weight of that component, based on the total weight of the composition of which the component is part.

5

For the purpose of this specification an amphiphile is defined as a compound which possesses an affinity for an apolar compound and for a polar compound. Suitably it forms a loose association in a composition, coupling
10 together other components of the composition, without forming a strongly micellar or lamellar structure, in the manner of a classical surfactant. Preferably it does not associate strongly with surfaces.

15 Preferably the water and the amphiphile co-exist without separating from each other, as layers. The amphiphile may suitably be dispersed in the water or in the form of droplets in the water, for example as in emulsion or a micro-emulsion. However the amphiphile and
20 water may alternatively separate from each other as layers, one layer preferably being a thin layer (the composition being adjacent to the phase transition line on the phase diagram). This layer may be a water-rich layer or, preferably, an amphiphile-rich layer

25

Preferably, the aqueous composition (as a single phase or as the aqueous layer, when there is separation of layers) has a water content of at least 90%, more preferably at least 94%, most preferably at least 96%, and
30 especially at least 98%.

Preferably the aqueous composition (as a single phase or as the aqueous layer, when there is separation of

layers) contains at least 1.5% of an amphiphile, more preferably at least 2%, most preferably at least 2.5%.

Preferably the aqueous composition (as a single phase or as the aqueous layer, when there is separation of layers) contains up to 20% of an amphiphile, more preferably up to 8%, most preferably up to 4%.

Suitable amphiphiles include substituted pyrrolidones, especially 1-alkyl-2-pyrrolidones. Preferably the alkyl group has, on average, 4 - 16 carbon atoms, more preferably 6 - 14 carbon atoms, and most preferably 8 - 12 carbon atoms. Preferably the alkyl group is linear.

Suitable amphiphiles include glycol ethers.

Preferred glycol ethers are compounds of the formula $R^1O(RO)_nH$ (I) in which R is a C_1-C_8 alkylene group (preferably C_1-C_4), n is at least 1 (preferably 2-4) and R^1 is a C_1-C_8 alkyl group (preferably C_1-C_4) or, especially, an optionally substituted aryl group). A preferred optionally substituted aryl group is an optionally substituted phenyl group. Substituents of a aryl or phenyl group include C_1-C_4 alkyl groups, C_1-C_4 alkoxy groups, C_1-C_4 haloalkyl groups, cyano groups, amido groups, amine groups, and halogen atoms. Preferred halogen atoms, including comprised within haloalkyl groups, include fluorine, chlorine and bromine atoms. There may suitably be 1-3 substituents. Preferably, however, an aryl or phenyl group is unsubstituted.

Specific examples of suitable glycol ethers not having an aromatic group include:

ethoxypropoxypropanol (EDP)
ethoxyethoxypropanol
propoxyethoxypropanol
propoxypropoxypropanol
5 butoxypropoxyethanol
butoxybutoxyethanol
butoxyethoxyethanol (BDGE)
ethoxypropanol (EP)
butoxyethanol (BE)
10 butyldiglycolether.

Specific examples of suitable, and preferred, glycol
ethers having an aromatic group include:

ethylene glycol phenyl ether (EPH)
15 phenoxypropanol (PhP)
phenoxypropoxypropanol (PhDP).

Especially preferred amphiphiles are compounds which
at no concentration are classified as VOCs as set out in
20 the regulations of the US Government Environmental
Protection Agency (EPA) for the envisaged application
(preferably as cleaners in a household environment) at the
priority date of this patent application. Other preferred
amphiphiles are compounds which are classified as VOCs by
25 the EPA regulations but only at particular concentration
levels; and which are present below such levels, in the
composition of the invention.

If water and an amphiphile of use in the present
30 invention are shaken together in a two-phase zone of the
phase diagram, away from a phase boundary, two easily-
visible phases will form. In some cases the water-rich
phase will be below the amphiphile-rich phase (amphiphiles

A). In other cases the water-rich phase will be above the amphiphile-rich phase (amphiphiles B). Provided that the proportions can be adjusted so that the composition is in accordance with the first aspect, the use of all such
5 amphiphiles A and B, is covered. Preferably, however, the amphiphiles used in this invention are of the type B.

Especially preferred glycol ethers are compounds having a vapour pressure of less than 0.1mmHg at 20°C.

10

Preferably the aqueous composition further contains a hydrocarbon. Preferably it contains at least 0.005% of a hydrocarbon, more preferably at least 0.01%, and most preferably at least 0.05%. Preferably it contains up to
15 5% of a hydrocarbon, more preferably up to 2%, yet more preferably up to 0.8%, and most preferably up to 0.5%. In preferred systems the hydrocarbon is present in an amount such that it separates from the composition to form a thin layer, preferably on top of the composition. In such
20 systems the amphiphile preferably does not also separate out. However the thin layer is generally predominantly hydrocarbon, with a minor concentration of amphiphile and water. The definitions given above for the amount of the hydrocarbon present include any separated-out hydrocarbon
25 that may form.

The hydrocarbon is suitably a paraffinic, including isoparaffinic compound. The hydrocarbon may suitably be a hydrocarbon fragrance. Preferably it is a liquid under
30 ambient conditions. Preferably the hydrocarbon has 5 or more carbon atoms, more preferably 8 or more. Preferably the hydrocarbon has up to 15 carbon atoms, more preferably

up to 12 carbon atoms. When the hydrocarbon is a mixture, as will often be the case, these definitions still apply, as mean values.

5 When a hydrocarbon is present it tends to be lighter than any other phase, and at certain hydrocarbon levels may separate out.

10 Preferably a hydrocarbon present is significantly more volatile than the water or the amphiphile.

15 In this specification when we mention an amphiphile or a hydrocarbon it is to be understood that there may be more than one of a said amphiphile or a hydrocarbon present. The weight percentage definitions which follow denote the total complement of amphiphile or hydrocarbon compounds present.

20 Suitably the composition may contain an electrolyte, preferably an ionic salt. Suitable examples include alkali metal salts, especially halides. Most preferred, for reasons which include cost and safety, is sodium chloride.

25 An electrolyte may be used to alter the phase diagram, shifting phase boundaries so that the composition is on or adjacent to a phase boundary, as required by the first aspect of the present invention.

30 Suitably the composition may contain a glycol. When it does so, the glycol is preferably present in an amount up to 10%, preferably up to 5%. Suitable glycols have up to up to 12 carbon atoms. Examples include 2-methyl-2,4-pentanediol and hexylene glycol.

Preferred compositions of the present invention do not contain hydrogen peroxide. Preferred compositions of the present invention do not contain any bleaching compounds
5 or bleach precursors or initiators.

Preferred compositions of the present invention do not contain a biostatic agent.

10 Remarkably, we have found that compositions of the invention may apparently exhibit behaviour of the Marangoni type. When a jar of a preferred composition of the invention is opened and exposed to the environment the surface may be seen to "twitch". This effect is believed
15 to be due to surface tension gradients, perhaps allied to crossing of phase boundaries, when volatile compounds evaporate. We have observed such compositions in which, when the lid is replaced on the jar, the "twitching" stops. The "twitching" may be observed in a preferred
20 embodiment if the composition is pipetted onto a surface. In a preferred embodiment in which a hydrocarbon is present and separates as a thin film on the surface of the composition, it is observed that when an aliquot of the hydrocarbon-rich phase is removed and pipetted onto a
25 surface, this exhibits the "twitching" behaviour.

By "twitching" we mean that the surface of the composition exhibited motility not induced by an external agent, such as an object applied to it; in other words a
30 self-induced motility.

In accordance with a second aspect of the present invention there is provided a cleaning composition

comprising water, a hydrocarbon and an amphiphile, which composition exhibits Marangoni behaviour when exposed to air. Preferably the composition exhibits Marangoni behaviour only when exposed to air.

5

For the purpose of this specification the Marangoni effect may be defined as the effect in a liquid when surface tension gradients cause movement of the liquid. The surface tension gradients may result from concentration changes (Gibbs effect), which may themselves arise from evaporation, or from inadequate mixing, folling separation or segregation in a container, or by the application of a stress, for example a shear force (as by a wiping action).

15

The Marangoni effect may give rise to cleaning benefits. Compositions exhibiting the Marangoni effect may exhibit a tendency to "creep" into small spaces. Evaporation of a compound may lead to changes in surface tension and to surface motility, causing the composition to move, for example into and then out of a crack, drawing soils with it. The use of amphiphiles may assist separation of dirt from a substrate. Amphiphiles generally do not promote adhesion with a surface but they may wet well. Thus amphiphile-containing compositions which exhibit the Marangoni effect may move into small interstices or may move between a surface and a soil deposit, "lifting" the latter.

25

The composition of the second aspect may be an aqueous composition, under if so the definitions given above in relation to the first aspect may be applied to it.

30

The composition of the second aspect may be a hydrocarbon-rich composition, which contains at least 96% of hydrocarbon, preferably at least 98%, most preferably at least 99%. Preferably it contains up to 99.99% of hydrocarbon, preferably up to 99.9%. The hydrocarbon may be as defined above in relation to the first aspect. Preferably the hydrocarbon-rich composition contains at least 0.1% of an amphiphile, more preferably at least 0.5%, most preferably at least 1%. Preferably it contains up to 4% of an amphiphile, more preferably up to 2.5%, most preferably up to 1.5%. The amphiphile may be as defined above in relation to the first aspect. Preferably hydrocarbon-rich composition contains at least 0.0001% of water, more preferably at least 0.001%. Preferably it contains up to 1% of water, more preferably up to 0.5%, most preferably up to 0.1%.

The composition of the second aspect may be an amphiphile-rich composition which contains at least 60% of an amphiphile, preferably at least 70%, most preferably at least 80%. Preferably it contains up to 96% of an amphiphile, preferably up to 92%, most preferably up to 80%. The amphiphile may be as defined above in relation to the first aspect. Preferably the amphiphile-rich composition contains at least 0.5% of hydrocarbon, more preferably at least 2%, most preferably at least 3%. Preferably it contains up to 10% of hydrocarbon, more preferably up to 8%, most preferably up to 6%. The hydrocarbon may be as defined above in relation to the first aspect. Preferably the amphiphile-rich composition contains at least 2% of water, more preferably at least 4%, most preferably at least 8%. Preferably it contains up

10

to 20% of water, more preferably up to 16%, most preferably up to 12%.

The composition of the second aspect may have a hydrocarbon-rich composition as the sole phase, an aqueous composition as the sole phase or an amphiphile-rich composition as the sole phase; in each case preferably as just set out. Alternatively it may be a composition in which two of them is present, or in which each of them is present, as a separate layer. Preferred three-layer compositions of the present invention have the amphiphile-rich layer as their bottom layer, and the aqueous layer as the middle layer, and the hydrocarbon layer as the top layer.

15

A preferred hydrocarbon-rich composition, isolated, is one which could (and preferably would), with different proportions of the hydrocarbon, amphiphile and water, form the top layer of such a three-layer composition.

20

A preferred amphiphile-rich composition, isolated, is one which would (and preferably would), with different proportions of the hydrocarbon, amphiphile and water, form the bottom layer of such a three-layer composition.

25

A preferred aqueous composition, isolated, is one which would (and preferably would), with different proportions of the hydrocarbon, amphiphile and water, form the middle layer of such a three-layer composition.

30

A particularly preferred composition of the present invention is one which is in accordance with both the

first aspect of the present invention and the second aspect of the present invention.

In accordance with a third aspect of the present invention there is provided a method of cleaning a soiled substrate, comprising the application to the soiled substrate of a composition of the first and/or second aspect of the present invention, as defined above.

10 The substrate may be, for example, a hard surface, for example, a surface in the kitchen or bathroom, including a work surface, floor, tile, glass, sanitaryware item, window, crockery item or a domestic appliance; or a fabric surface, for example a clothing or upholstery item.

15

The soiling on the substrate may, for example, be grease, a protein stain, wine, fruit juice or make-up.

Preferred compositions of the present invention are provided in containers which have a manually operable pump such as a "trigger spray", or in containers from which the contents are poured, or expressed by squeezing the container, or in aerosol dispensers. In aerosol dispensers evaporation of propellant could itself induce a Marangoni effect.

25

Containers containing ready-to-use compositions of the invention constitute a further aspect of the present invention, notably containers adapted to deliver such compositions directly to hard surfaces (for example aerosols and "trigger sprays"). Containers containing aqueous dilutable compositions constitute a still further aspect.

30

The invention will now be further described, by way of example, with reference to the following example.

- 5 In these examples the three materials studied were deionised water, isoparaffinic solvent ISOPAR E and, as glycol ether, DOWANOL EPh (ethylene glycol phenyl ether). ISOPAR E and DOWANOL EPh are Trade Marks.
- 10 First, to "scope" the work the three materials were mixed together in different proportions and allowed to separate. As a result the phase diagram shown in Figure 1 was plotted. To our surprise, in the large, triangular central region of the ternary phase diagram, denoting the
- 15 presence of three distinct phases, it was the lower phase which was the glycol ether-rich phase, and the middle phase which was the water-rich phase. This was different to the finding presented in EP 330379A.
- 20 It will be seen that the phase diagram shows phase changes at each corner, the water-rich corner being the region of greatest interest to us. Small changes in the composition, for example induced by preferential evaporation of one component, are likely to cause the
- 25 composition to cross one or more phase boundaries. This is believed to be of significance in the findings that we report below.
- 30 A representative material having three distinct layers region was analysed as follows, for the content of each layer. The total of each layer does not come to exactly 100%, due to experimental tolerance. However there were no further materials present.

Top Layer - ISOPAR E-rich layer

Water level = less than 0.1% (average of 3 readings)
ISOPAR E level = 99.8% (average of 3 readings)
5 DOWANOL EPh level = 1.2% (average of 3 readings)

Middle Layer - Water-rich layer

Water level = 96.2% (average of 6 readings)
ISOPAR E level = less than 0.1% (average of 3 readings)
10 DOWANOL EPh level = 2.6% (average of 3 readings)

Bottom Layer - EPh-rich layer

Water level = 9.4% (average of 3 readings)
ISOPAR E level = 4.3% (average of 3 readings)
15 DOWANOL EPh level = 85.3% (average of 3 readings)

Based on this analysis and our desire to aim for an aqueous cleaning composition, a series of test compositions were formulated based on the aqueous
20 composition, corresponding to the middle layer, as set out in the table below:

COMPOSITION NO.	CONCENTRATION OF INGREDIENTS			OBSERVATIONS
	WATER (g)	ISOPAR E (g)	DOWANOL EPH (g)	
1	96.2	0.11	2.6	2 phases. 2mm layer on top of aqueous layer
2	9.625	0.015	0.261	Clear, 1 phase, with oil drops on top
3	9.73	0.013	0.261	Clear, 1 phase
4	9.72	0.029	0.27	Clear, 1 phase with small drops at bottom

Any separated material on top of the aqueous composition is hydrocarbon-rich material, and that any composition separated at the bottom of the aqueous composition is glycol ether-rich material.

Each composition was then subjected to an assessment of its properties. A few drops of each composition was pipetted from the central region of a vial, without prior shaking, onto a clean laboratory bench top and observed for "twitching" behaviour. Subsequently, a few drops of each composition was applied to pencil marks on a horizontal enamel surface, and after a period removed by gentle wiping. The results were as follows:

COMPOSITION NO.	OBSERVATIONS	
	"TWITCHING"	CLEANING
1	Surface in vial "twitched". Stayed still on bench	Cleaned pencil marks
2	Surface in vial "twitched". Stayed still on bench	Cleaned pencil marks
3	Stayed still in vial and on bench	Cleaned pencil marks
4	Surface in vial "twitched". Stayed still on bench	Cleaned pencil marks
Deionised water (comparison)	---	Did not clean pencil marks

In a further test composition no. 1 was assessed more closely. It was observed that the thin top layer, having risen to cover the aqueous layer, "twitched" excessively in the vial, when open for 10 minutes. The "twitching" terminated when the vial was stoppered.

When composition no. 1 was pipetted onto the bench careful observation showed that a top film separated, and "twitched". We believe that this Marangoni-type activity was associated with evaporation of the ISOPAR E.

In another test we also carefully pipetted an aliquot of the thin top layer of composition no. 1 and spread this on top of a clean laboratory worktop. This composition also twitched, and it was also observed that it spread out itself, by a "creeping" action.

In a further experiment ISOPAR E was dropped onto the deionised water in a beaker, stirred to mix, and allowed to separate as a surface layer. It was observed that the system did not twitch. We inferred (with caution, for
5 this was only a preliminary test) that it might be necessary for the glycol ether to be present for the "twitching" phenomenon to occur, even though it is only present in small amounts in the aqueous phase and in the hydrocarbon-rich phase.

10

In a further experiment, when 0.26 g of DOWANOL EPh was added to 9.62 g of deionized water, with no ISOPAR E present, it was observed that the resulting solution appeared itself to show Marangoni behaviour; the resulting
15 solution appeared to "climb" up the side of the beaker.

Further tests were carried out, to test the possible effectiveness of composition no. 1 against stains on fabrics. In these tests approximately 100ml of
20 composition number 1 was pipetted onto a cellulosic pad, and pressed against a proprietary bought-in stained substrate, with another cellulosic pad, dry, underneath the stained substrate (so that the two cellulosic pads formed a sandwich, about the stained substrate. A heated
25 iron (hot plate temperature 167 °C) was applied to the moistened pad. In this manner composition no. 1 was found to be effective at removing red wine staining from cotton fabric, grass staining from cotton fabric, make-up staining from silk fabric, and red wine staining from silk
30 fabric. The cleaning action was to "drive" the respective stain through to the dry pad underneath.

Figure 2 shows the phase diagram for another system of interest in this invention, 1-(n-octyl)-2-pyrrolidone / n-dodecane / water. It will be seen that this phase diagram also shows phase changes at the water-rich corner, moving
5 from two phases (1-(n-octyl)-2-pyrrolidone / water) to three phases, to two phases (n-dodecane / water). Small changes in the composition, for example induced by preferential evaporation of one component, are again likely to cause the composition to cross a phase boundary.
10 We believe this is of significance in this system in obtaining a "twitching" effect.

Claims

1. A cleaning composition comprising at least 80% w/w water and at least 1% w/w of an amphiphile, the composition being on or adjacent to a phase boundary on the phase diagram for the composition.
5
2. A cleaning composition as claimed in claim 1, wherein the water and the amphiphile co-exist without separating from each other as layers.
10
3. A cleaning composition as claimed in claim 1 or 2, wherein the composition contains at least 96% w/w water.
15
4. A cleaning composition as claimed in any preceding claim, wherein the composition contains 2 - 8% w/w of amphiphile.
- 20 5. A cleaning composition as claimed in any preceding claim, wherein the amphiphile is a glycol ether.
6. A cleaning composition as claimed in claim 5, wherein the glycol ether is a compound of the
25 formula $R^1O(RO)_nH$ (I) in which R is a C_1-C_8 alkylene group, n is at least 1 and R^1 is a C_1-C_8 alkyl group, or is an optionally substituted aryl group.
- 30 7. A cleaning composition as claimed in claim 6, wherein R^1 is a phenyl group.

8. A cleaning composition as claimed in any of claims 1 to 4, wherein the amphiphile is a 1-substituted-2-pyrrolidone.
- 5 9. A cleaning composition as claimed in any preceding claim, wherein the amphiphile is of a type such that, when it is mixed with water in a proportion to form two phases, the water-rich phase is above the amphiphile-rich phase.
- 10 10. A cleaning composition as claimed in any preceding claim, wherein the aqueous composition further contains a hydrocarbon.
- 15 11. A cleaning composition as claimed in claim 10, wherein the hydrocarbon is present in an amount in the range 0.005 - 5 % w/w, preferably 0.05 - 0.5 % w/w.
- 20 12. A cleaning composition as claimed in claim 11, wherein the hydrocarbon is in an amount such that it separates from the composition to form a thin layer on top of the composition.
- 25 13. A cleaning composition comprising water, a hydrocarbon and an amphiphile, which composition exhibits Marangoni behaviour when exposed to air.
- 30 14. A cleaning composition as claimed in claim 13, which composition is a hydrocarbon-rich composition, an amphiphile-rich composition or a water-rich composition; preferably a water-rich composition.

15. A method of cleaning a soiled substrate, comprising the application to the soiled substrate of a composition as claimed in any preceding claim.

5

16. A cleaning composition or a method of cleaning, substantially as hereinbefore described with particular reference to the accompanying drawings.

ABSTRACT**Composition and Method**

5 A cleaning composition comprising a major amount of water
a minor amount of an amphiphile (preferably a glycol
ether) and optionally a minor amount of a hydrocarbon has
been shown to exhibit interesting cleaning performance.
Preferably the composition is on or adjacent to a phase
10 boundary of its phase diagram. Compositions of the
present invention have been shown to exhibit self-induced
surface motility of the Marangoni type.

15

(not recommended to publish with a drawing)

